

REPORT OF COMPLIANCE BIOMONITORING INSPECTION-ACUTE

AT

DOE RUN HERCULANEUM SMELTER

HERCULANEUM, MISSOURI

NPDES PERMIT NUMBER: MO-0000281

FEBRUARY 22-24, 1999

BY

U.S. ENVIRONMENTAL PROTECTION AGENCY

Region VII

Environmental Services Division

0717

| | |
|--------|---------------|
| Site: | Herculaneum |
| ID #: | MO0000281-873 |
| Break: | LO |
| Other: | 2/22/99 |

INTRODUCTION

At the request of the Water, Wetlands and Pesticides Division, NPDES Facilities Management Branch, I performed a Compliance Biomonitoring Inspection-Acute (CBI-A) of the Doe Run Herculaneum Smelter in Herculaneum, Missouri on February 22-24, 1999. This narrative report and attachments present the results of the inspection.

PARTICIPANTS

Doe Run Herculaneum Smelter:

James M. Lanzafame, Environmental Manager

U.S. Environmental Protection Agency (EPA):

Lorenzo P. Sena, Environmental Protection Specialist

Bruce A. Littell, Environmental Scientist

INSPECTION PROCEDURES

Facility personnel were not notified prior to the inspection. Upon arrival to the facility Mr. Littell and I contacted Mr. Lanzafame from the lobby, Mr. Lanzafame arrived shortly after. We presented our credentials and explained the purpose and procedures of this inspection. We then set up an appointment for February 24, 1999. Mr. Lanzafame escorted Mr. Littell and I to Outfall 001 where we set up an ISCO 1580 HS wastewater sampler. This CBI-A consisted of the review of facility records, sample collection and sample analysis, and an inspection of the facility. On February 24, Mr. Lanzafame escorted Mr. Littell and I on a tour of the facility.

40199145



SUPERFUND RECORDS

FACILITY DESCRIPTION

The Doe Run Company is a lead manufacturing company which owns and operates several mines, mills and smelters. There are eight mines (all located in the Viburnum Trend), five mills and three smelters all of which are located in eastern Missouri. The Herculanum smelter is also a lead refinery. This facility occupies 26 to 28 acres of the 620 acres owned by Doe Run in the area. The lead products manufactured at this facility are derived from galena (PbS) ore concentrate.

The ore concentrate is transported by rail or truck from one of the five Doe Run mills. The lead concentrate is comprised of about 78% lead sulfide (PbS) and 12-14% sulphur (S). The concentrate is mixed with sand, hematite and secondaries until the concentration of PbS is approximately 45%. This mixture is fed into the sintering machine. Sintering converts the lead sulfide into lead oxide and produces sulphur dioxide, which is used to produce sulfuric acid. Approximately 330,000 tons/year of lead concentrate is processed. Finish sinter is the product of the sintering operation.

Finish sinter is fed to the blast furnaces. Coke, oxygen and blasting air are used to reduce the lead oxide to lead bullion. The facility operates an oxygen plant to supply the furnaces with oxygen for reduction. There are three blast furnaces in this operation two of which are used at any one time. Lead bullion contains mostly lead along with some impurities such as copper, silver and zinc. The primary purpose of the dross plant is the removal of copper. Sulfur and pyrite are added to the bullion to tie up copper in the lead bullion. As the bullion cools, the copper floats to the surface as copper dross. The copper dross is skimmed off and sold as a byproduct.

Decopperized bullion is then transferred to the refinery, in which there are 11 kettles. The refining process consists of multiple steps used to take out silver and zinc, producing 99.99 - 99.999% pure lead. The lead is then cast into 1 ton ingots, 65 pound pigs and 100 pound pigs. A specialty product called lead strip is also made and is primarily for the battery making industry. In 1998 approximately 250,000 tons of lead products were manufactured. Doe Run also formulates lead alloys to customer specifications.

Doe Run not only produces lead but to a lesser extent produces copper dross, silver dross and sulfuric acid. Sulfuric acid is produced as a byproduct of the oxidation of the lead ore concentrate. A Monsanto acid plant, which was constructed in 1969, is used for this process. A new stack was added to the acid plant in September of 1997. There are four designated acid tanks at the north end of the facility (Figure 1) one of which is only used as an emergency overflow tank, and has a capacity of 1,000,000 gallons. The remaining three storage tanks are for acid storage and have capacities of 600,000 gallons, 244,000 gallons and 244,000 gallons, respectively. There is also a product tank located near the acid plant and it has a capacity of 244,000 gallons. These are tank capacities however and do not reflect actual volumes of acid in storage. Sulfuric acid production is roughly 60,000 to 70,000 tons per year which is shipped by barge with smaller amounts by truck and rail.

The Herculanum smelter currently employs 450 people 90 of which are contractors. The plant operates 24 hours per day and about 355 days per year. There is a scheduled shutdown every year for construction and major repairs, this shutdown lasts one to two weeks.

AIR EMISSIONS

Doe Run Herculanum Smelter analyzes ambient sulfur dioxide continuously and lead is measured routinely on an every sixth day schedule. New fans were installed in baghouse #3 and are larger than the fans previously used, Mr. Lanzafame also mentioned that they were going to start using Teflon bags. According to Mr. Lanzafame, Teflon bags decrease pressure drop within the baghouse and at the same time collect smaller particles and have a longer bag life. Baghouse #3 has been in operation since 1957.

WASTEWATER TREATMENT

Done sinter is stored outside on concrete pads at the south end storage area (Figure 1). Runoff from this paved area is collected in drains and is routed to the wastewater treatment plant. Approximately 100 meters south of the south end storage area is a zinc-bearing slag pile. It was explained that this slag is not considered a hazardous waste. It is comprised of magnesium, silica, iron and about 10- 14% zinc. Runoff from this area is not contained or treated in any way. A storm water permit is pending with the state of Missouri. We were told that this slag is kept because in the future they plan to extract the zinc and sell the zinc metal as product.

Water used in the manufacturing process at the Herculanum facility is primarily drawn by three pumps at the Ranney well (Figure 1). In addition approximately 140 gallons per minute of city water is used for bearing cooling water and for all personal use. Sanitary wastewater is sent to the Herculanum Sewer District waste water treatment plant.

Attachment 1 outlines the facility processes which use water. Process water along with storm water drainage from the paved areas of the facility are sent to a sump in the flux building (Figure 2) at a pH of 5 to 7. Process water and storm water are then pumped to the south tank (Item 8 in Figure 1). From the south tank water is pumped to the equalization/thickener (ET) tank at the wastewater treatment area. From the ET tank a portion of the water is returned to the north tank (Figure 2).

Part of the re-circulated water from the equalization/thickener (ET) tank passes through the wastewater treatment plant before it is released to the Mississippi River. From the ET tank the water enters the reactor where lime and polymers are added. The water proceeds through the reactor clarifier and then through three municipal grade sand-anthracite filters in parallel. Treated water is then piped to a small cement structure, where it flows over a 90 degree V-notch weir before entering the Mississippi River as Outfall 001. An ultrasonic detector is used to measure flow and is coupled to a totalizer. Treated water can also be returned to the plant water system by opening a manual valve at the weir-box (Figure 2).

Clarifier sludge is thickened in the equalization plant prior to being dewatered in a large plate and frame filter press. Filtrate is then returned to the headworks at the ET tank. Dewatered sludge is loaded into a railcar and added to the sintering operation along with baghouse dust (which is approximately 60% lead). The waste water treatment plant is staffed by an operator 24 hours/day and 365 days/year.

The purpose of Outfall 002 is to release excess water in the event of a malfunction of the flux building sump pumps, or if their capacity is exceeded. Outfall 002 can only be discharged if two bolted on emergency caps are manually removed. Outfall 002 begins as a standpipe in the flux building sump and is of sufficient height to cause the sump building to flood which would alert personnel of a problem. The standpipe was installed in 1994. At Outfall 002 there is an 8 inch tall and 2 foot wide rectangular weir (Figure 1) for measuring instantaneous flow. In addition, an ISCO Model 2700 composite sampler and a flow meter are available for use in the event of a discharge. No discharge was observed at Outfall 002 during the inspection.

There is a direct line from the Ranney well to the acid plant. Outfall 003 is once-through cooling water from the acid plant. The flow at Outfall 003 is measured with a McCrometer, Inc. in-line turbine meter coupled to a totalizer. The totalizer is read daily.

There are no provisions for emergency power in the event of a power failure, Mr. Lanzafame explained that when the power shuts off, everything at the facility stops, including wastewater flow.

Water parameters in the Doe-Run Herculaneum Smelter permit, with the exception of pH, temperature and total suspended solids, are analyzed at their Viburnum facility.

SAMPLE COLLECTION AND ANALYSES

I installed an ISCO Model 1580 HS automatic sampler at the weir box of Outfall 001, adjacent to the composite sampler used by the Doe Run Herculaneum Smelter sampling personnel.

The sampler was equipped with new pump and plastic intake tubing which meets sampling requirements, and a pre-cleaned three-gallon polyethylene container for sample collection. I rinsed the tubing and collection container with effluent water prior to use. I then placed the sample container in the sampler and iced it. I set the sampler to take a 224 mL aliquot every 30 minutes for 24 hours.

During the period of February 22-23, 1999, I collected a 24 hour composite sample from Outfall 001 in accordance with approved biomonitoring sampling protocol. I also collected grab samples from Outfall 001 and 003 on February 23, 1999 for metals, pH and temperature analysis. Mr. Lanzafame collected grab samples at the same time. There was no discharge observed at Outfall 002.

All samples were collected and preserved in accordance with standard operating procedures and with the exception of pH and temperature (which were determined on-site), were shipped via Federal Express to the EPA, Region VII Laboratory for analysis using approved chain-of-custody and tamper evident seals.

FINDINGS AND OBSERVATIONS

The following findings and observations were noted during the sampling, plant and record review. A complete summary of review is given in the NPDES Compliance Inspection Report (Form 3560-3, Attachment 2).

1. During the laboratory record keeping review, equipment calibration logs were reviewed. During the previous inspection it was noted that the lab was not performing pH meter calibration according to EPA accepted methods (two point calibration method using two buffers of known pH). A laboratory operator demonstrated how the pH meter is now being calibrated using the two point method. Upon review of the laboratory bench sheets (which show date of collection, time of collection and time of analysis), Mr. Littell noted that the sample holding time for pH analysis was frequently greater than 15 minutes.
2. During the interview with Mr. Lanzafame I noted that the SIC code on the permit (3332) is incorrect, the correct SIC code is 3339 (Primary smelting and refining of nonferrous metals, except copper and aluminum).
3. During review of the DMR data, pH exceedances were observed for Outfall 001, on 11/28/1998 (pH 10.62) and also on 11/30/1998 (pH 10.78). I viewed a copy of the letter mailed to the state. According to the letter, the reason for the exceedances was a malfunctioning pH probe. The pH probe was replaced.
4. Effluent from Outfall 001 was shown to be acutely toxic to both C. dubia and P. promelas. All thirty P. promelas exposed to the 100% effluent, fifteen out of thirty in the 50% and eight out of the thirty in the 25% effluent (all statistically significant) died by the end of the 48 hour test. The 48 hour LC_{50} value for P. promelas exposed to the Doe-Run Herculanum Smelter effluent was calculated to occur at 39.7% effluent. Significant mortality was also observed for C. dubia, as eight out of the twenty in the 100% effluent were dead after 48 hours of exposure. No mortality was observed at the lower concentrations. The 48 hour LC_{50} for C. dubia could not be calculated since less than 50% mortality was observed (Attachment 3). According to the permit special conditions the Allowed Effluent Concentration (AEC) is 10%, in which case no toxicity would have been observed (Attachment 4).
5. The grab samples taken from Outfalls 001 and 003 were tested for the metals specified in the permit. The results (Attachment 5) showed that the effluent samples were within permit limits.

6. Surface runoff from the facility flows into floor drains which report to the flux building sump. It is then pumped to the waste water treatment plant. A bypass was reported to the state on June 6, 1997. The reason for the bypass was excessive rainfall which caused the flux building pump capacity to be exceeded.

7. In addition to the CBI-A, I also completed the attached EPA Region VII Level B Multimedia Screening Checklist (Attachment 6). Copies of the checklist were forwarded to the appropriate programs.

RECOMMENDATIONS

1. Laboratory and sample collection practices must be altered to reduce holding time for pH analysis, in order to conform with 40 CFR 136.3, as discussed in the inspection.

Bruce Jittell (For LPS)

Lorenzo P. Sena

Environmental Protection Specialist

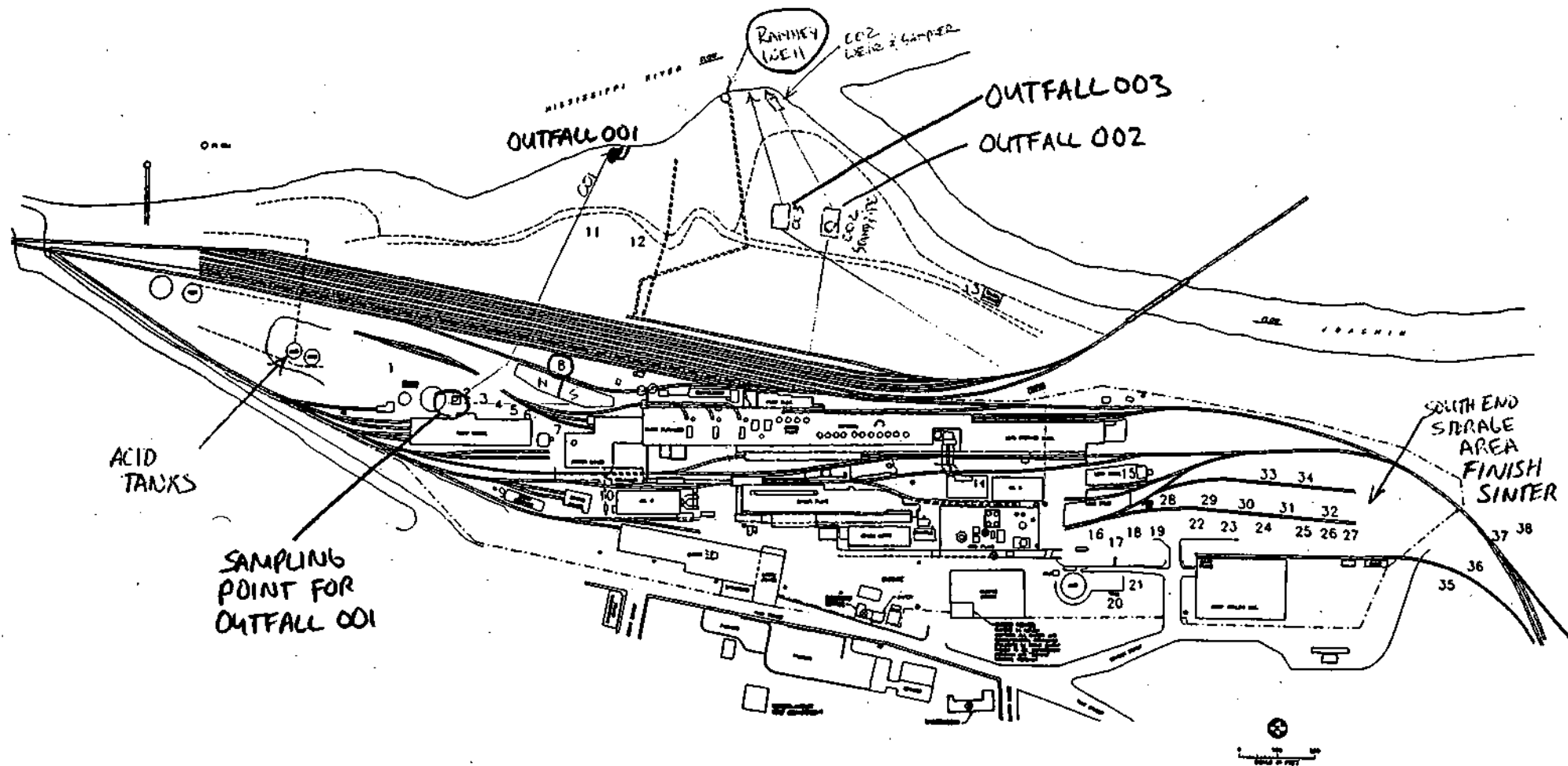
Activity Number: WJF46

Date: 4/20/99

ATTACHMENTS

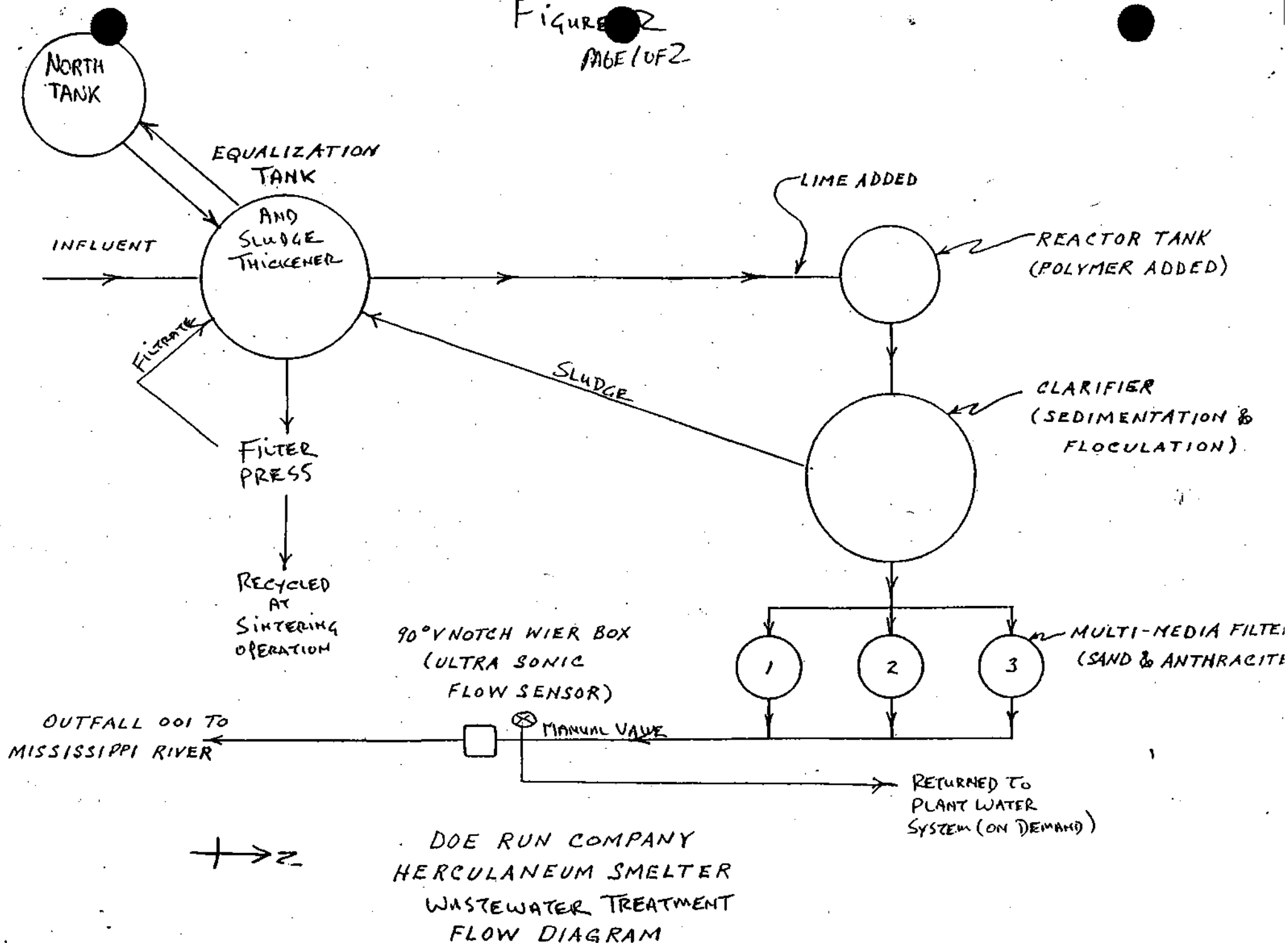
1. Sources of Water Reporting to Doe Run Herculaneum Smelter Wastewater Treatment Plant (1 page).
2. Water Compliance Inspection Report EPA Form 3560-3 (4 pages).
3. Toxicity Test Results (7 pages).
4. Analytical Request Report (5 pages).
5. Missouri State Operating Permit, #MO-0000281 (8 pages).
6. Region VII Multimedia Screening Checklist (2 pages).

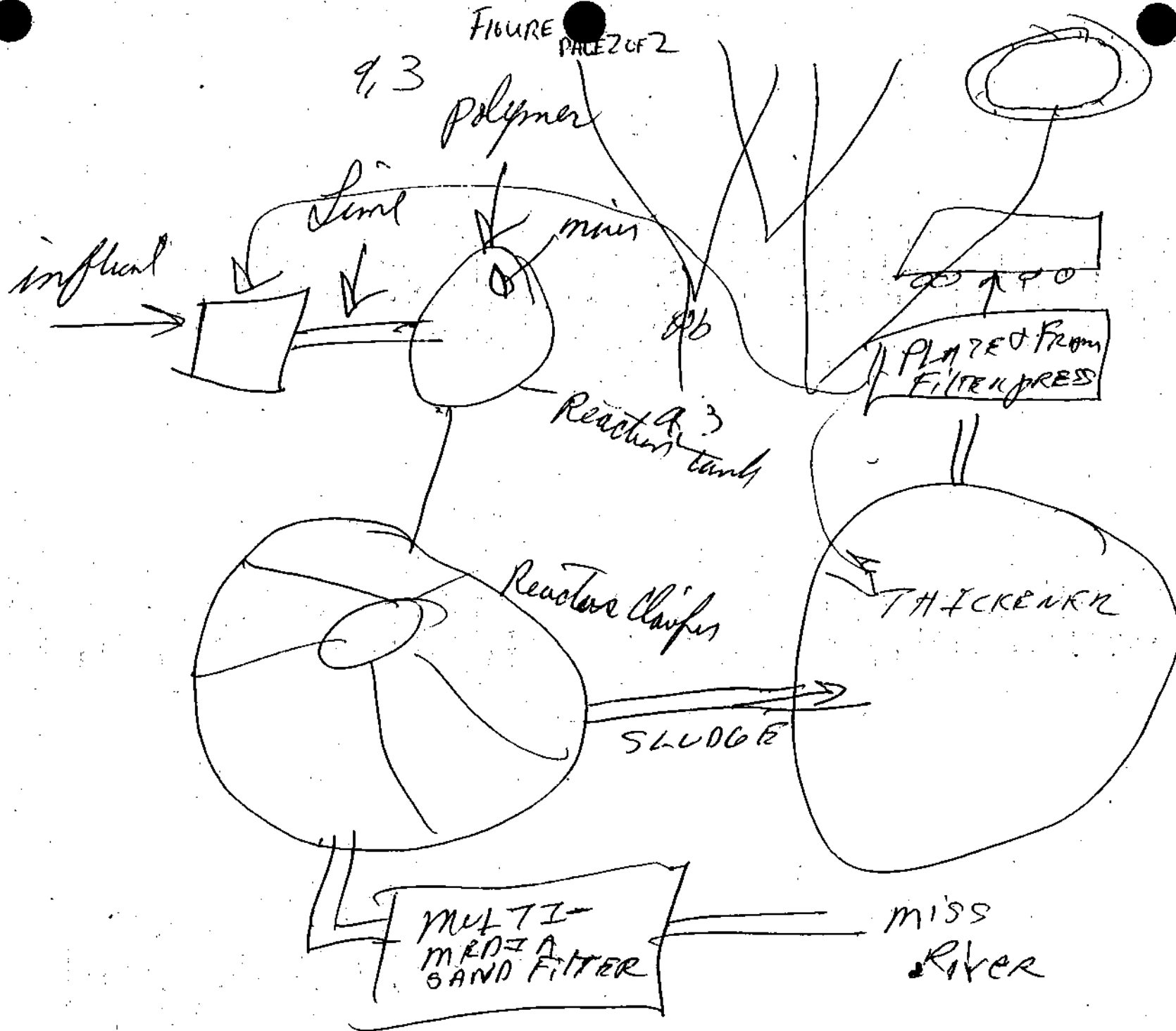
FIGURE 1



| | |
|-------------------|--|
| DOE RUN | |
| PLANT & EQUIPMENT | |
| PROPERTY LINE | |
| DATE | |
| DRAWN BY | |
| CHECKED BY | |
| APPROVED BY | |
| DATE | |

Figure 2
PAGE 1 OF 2





End

ATTACHMENT 1

List of wastewater sources reporting to the Doe-Run Herculanum Smelter wastewater treatment plant

1. Plant washdown of pavement and rainfall
2. Departmental washdown throughout plant
3. Laundry uniform wash water
4. Employee hand wash water
5. Respirator wash water
6. Sinter plant conveyor belt water
7. Acid plant blowdown
8. Acid plant cooling tower blowdown
9. Acid plant bearing cooling water
10. Baghouse bearing cooling water
11. Blast furnace slag granulation water blowdown
12. Blast furnace cooling tower blowdown
13. Blast furnace conveyor belt water
14. Dross plant dross wetting water
15. Wet dross drainage
16. Dross furnace cooling tower blowdown
17. A portion of the refinery casting water
18. Refinery cooling tower blowdown
19. Sludge filtrate
20. Sand Vertical Gravity filter (SVG) backwash water

Form Approved.
OMB No. 2040-0057
Approval expires 8-31-98

Section A: National Data System Coding (i.e., PCS)

| Transaction Code | | NPDES | | yr/mo/day | | Inspection Type | | Inspector | | Fac Type | | | | | | | | | | | | | | |
|----------------------|---|-------|---|--|--------------|-----------------|-------|-----------|--|----------|---|----------|---|----|----|----|--|----|--|--|--|--|--|----|
| 1 | N | 2 | 5 | 3 | 101010102811 | 12 | 91902 | 17 | | 18 | B | 19 | R | 20 | 2 | | | | | | | | | |
| Remarks | | | | | | | | | | | | | | | | | | | | | | | | |
| 21 | | | | | | | | | | | | | | | 66 | | | | | | | | | |
| Inspection Work Days | | | | Facility Self-Monitoring Evaluation Rating | | | | B1 | | QA | | Reserved | | | | | | | | | | | | |
| 67 | | | | 69 | | | | 70 | | 71 | D | 72 | N | 73 | | 74 | | 75 | | | | | | 80 |

Section B: Facility Data

| | | | |
|---|--|---------------------------------|------------------------------------|
| Name and Location of Facility Inspected (For industrial users discharging to POTW, also include POTW name and NPDES permit number) DOE RUN-HERCULANEUM SMELTER 881 MAIN STREET HERCULANEUM, MO 63048 | | Entry Time/Date 2:30 2/22/95 | Permit Effective Date 11/09/95 |
| Name(s) of On-Site Representative(s)/Title(s)/Phone and Fax Number(s) JIM LANZAFAME / ENVIRONMENTAL MANAGER (314) 479-5311 FAX (314) 933-3150 | | Exit Time/Date 4:10 PM | Permit Expiration Date 03/22/00 |
| Name, Address of Responsible Official/Title/Phone and Fax Number SAME AS ABOVE | | Other Facility Data | |

Contacted
☒ Yes ☐ No

Section C: Areas Evaluated During Inspection (Check only those areas evaluated)

| | | | | | | | |
|-------------------------------------|---------------------------|-------------------------------------|-------------------------|-------------------------------------|--------------------------|-------------------------------------|--------------------------|
| <input checked="" type="checkbox"/> | Permit | <input checked="" type="checkbox"/> | Flow Measurement | <input checked="" type="checkbox"/> | Operations & Maintenance | <input type="checkbox"/> | CSO/SSO (Sewer Overflow) |
| <input checked="" type="checkbox"/> | Records/Reports | <input checked="" type="checkbox"/> | Self-Monitoring Program | <input checked="" type="checkbox"/> | Sludge Handling/Disposal | <input type="checkbox"/> | Pollution Prevention |
| <input checked="" type="checkbox"/> | Facility Site Review | <input type="checkbox"/> | Compliance Schedules | <input type="checkbox"/> | Pretreatment | <input checked="" type="checkbox"/> | Multimedia |
| <input checked="" type="checkbox"/> | Effluent/Receiving Waters | <input checked="" type="checkbox"/> | Laboratory | <input type="checkbox"/> | Storm Water | <input type="checkbox"/> | Other: |

Section D: Summary of Findings/Comments (Attach additional sheets of narrative and checklists as necessary)

| | | |
|--|---|---------|
| Name(s) and Signature(s) of Inspector(s) | Agency/Office/Phone and Fax Numbers | Date |
| LORENZO P. SENA / LORENZO P. SENA | EPA REGION 7 / FMILC (414) 551-5014 (414) 551-5214 | 2/23/99 |
| | | |
| | | |
| | | |
| Signature of Management Q A Reviewer | Agency/Office/Phone and Fax Numbers | Date |
| | | |

Sections F thru L: Complete on all inspections, as appropriate. N/A = Not Applicable

PERMIT NO.

Mo 0000281

SECTION F - Facility and Permit Background

ADDRESS OF PERMITTEE IF DIFFERENT FROM FACILITY
(Including City, County and ZIP code)

DATE OF LAST PREVIOUS INVESTIGATION BY EPA/STATE

FINDINGS

SECTION G - Records and Reports

RECORDS AND REPORTS MAINTAINED AS REQUIRED BY PERMIT.

☒ YES ☐ NO ☐ N/A (Further explanation attached _____)

DETAILS:

(a) ADEQUATE RECORDS MAINTAINED OF:

(i) SAMPLING DATE, TIME, EXACT LOCATION

☒ YES ☐ NO ☐ N/A

(ii) ANALYSES DATES, TIMES

☒ YES ☐ NO ☐ N/A

(iii) INDIVIDUAL PERFORMING ANALYSIS

☒ YES ☐ NO ☐ N/A

(iv) ANALYTICAL METHODS/TECHNIQUES USED

☐ YES ☐ NO ☐ N/A

(v) ANALYTICAL RESULTS (e.g., consistent with self-monitoring report data)

☒ YES ☐ NO ☐ N/A

(b) MONITORING RECORDS (e.g., flow, pH, D.O., etc.) MAINTAINED FOR A MINIMUM OF THREE YEARS INCLUDING ALL ORIGINAL STRIP CHART RECORDINGS (e.g., continuous monitoring instrumentation, calibration and maintenance records).

☒ YES ☐ NO ☐ N/A

(c) LAB EQUIPMENT CALIBRATION AND MAINTENANCE RECORDS KEPT. ONLY DO PH, TSS

☒ YES ☐ NO ☐ N/A

(d) FACILITY OPERATING RECORDS KEPT INCLUDING OPERATING LOGS FOR EACH TREATMENT UNIT.

☒ YES ☐ NO ☐ N/A

(e) QUALITY ASSURANCE RECORDS KEPT. QUINCEBOS DONE VIBRATION FACILITY

☐ YES ☐ NO ☒ N/A

(f) RECORDS MAINTAINED OF MAJOR CONTRIBUTING INDUSTRIES (and their compliance status) USING PUBLICLY OWNED TREATMENT WORKS.

☐ YES ☐ NO ☒ N/A

SECTION H - Permit Verification

INSPECTION OBSERVATIONS VERIFY THE PERMIT.

☒ YES ☐ NO ☐ N/A (Further explanation attached _____)

DETAILS:

(a) CORRECT NAME AND MAILING ADDRESS OF PERMITTEE.

☒ YES ☐ NO ☐ N/A

(b) FACILITY IS AS DESCRIBED IN PERMIT.

☒ YES ☐ NO ☐ N/A

(c) PRINCIPAL PRODUCT(S) AND PRODUCTION RATES CONFORM WITH THOSE SET FORTH IN PERMIT APPLICATION.

☒ YES ☐ NO ☐ N/A

(d) TREATMENT PROCESSES ARE AS DESCRIBED IN PERMIT APPLICATION.

☒ YES ☐ NO ☐ N/A

(e) NOTIFICATION GIVEN TO EPA/STATE OF NEW, DIFFERENT OR INCREASED DISCHARGES.

☐ YES ☐ NO ☒ N/A

(f) ACCURATE RECORDS OF RAW WATER VOLUME MAINTAINED.

☐ YES ☒ NO ☐ N/A

(g) NUMBER AND LOCATION OF DISCHARGE POINTS ARE AS DESCRIBED IN PERMIT.

☒ YES ☐ NO ☐ N/A

(h) CORRECT NAME AND LOCATION OF RECEIVING WATERS.

☒ YES ☐ NO ☐ N/A

(i) ALL DISCHARGES ARE PERMITTED.

☒ YES ☐ NO ☐ N/A

SECTION I - Operation and Maintenance

TREATMENT FACILITY PROPERLY OPERATED AND MAINTAINED.

☐ YES ☐ NO ☐ N/A (Further explanation attached _____)

DETAILS:

(a) STANDBY POWER OR OTHER EQUIVALENT PROVISIONS PROVIDED. EVERYTHING SHUT WHEN POWER OFF.

☐ YES ☒ NO ☐ N/A

(b) ADEQUATE ALARM SYSTEM FOR POWER OR EQUIPMENT FAILURES AVAILABLE.

☐ YES ☒ NO ☐ N/A

(c) REPORTS ON ALTERNATE SOURCE OF POWER SENT TO EPA/STATE AS REQUIRED BY PERMIT.

☐ YES ☐ NO ☒ N/A

(d) SLUDGES AND SOLIDS ADEQUATELY DISPOSED. RECYCLED MANAGED

☒ YES ☐ NO ☐ N/A

(e) ALL TREATMENT UNITS IN SERVICE.

☒ YES ☐ NO ☐ N/A

(f) CONSULTING ENGINEER RETAINED OR AVAILABLE FOR CONSULTATION ON OPERATION AND MAINTENANCE PROBLEMS.

☒ YES ☐ NO ☐ N/A

(g) QUALIFIED OPERATING STAFF PROVIDED.

☒ YES ☐ NO ☐ N/A

(h) ESTABLISHED PROCEDURES AVAILABLE FOR TRAINING NEW OPERATORS.

☒ YES ☐ NO ☐ N/A

(i) FILES MAINTAINED ON SPARE PARTS INVENTORY, MAJOR EQUIPMENT SPECIFICATIONS, AND PARTS AND EQUIPMENT SUPPLIERS.

☒ YES ☐ NO ☐ N/A

(j) INSTRUCTIONS FILES KEPT FOR OPERATION AND MAINTENANCE OF EACH ITEM OF MAJOR EQUIPMENT.

☒ YES ☐ NO ☐ N/A

(k) OPERATION AND MAINTENANCE MANUAL MAINTAINED.

☒ YES ☐ NO ☐ N/A

(l) SPCC PLAN AVAILABLE.

☒ YES ☐ NO ☐ N/A

(m) REGULATORY AGENCY NOTIFIED OF BY PASSING. (Date: 10/17/97)

☒ YES ☐ NO ☐ N/A

(n) ANY BY-PASSING SINCE LAST INSPECTION.

☐ YES ☒ NO ☐ N/A

(o) ANY HYDRAULIC AND/OR ORGANIC OVERLOADS EXPERIENCED.

☐ YES ☒ NO ☐ N/A

PERMIT NO.

MO 0000281

SECTION J - Compliance Schedules

PERMITTEE IS MEETING COMPLIANCE SCHEDULE.

☐ YES☒ NO☐ N/A

(Further explanation attached _____)

CHECK APPROPRIATE PHASE(S):

- ☐ (a) THE PERMITTEE HAS OBTAINED THE NECESSARY APPROVALS FROM THE APPROPRIATE AUTHORITIES TO BEGIN CONSTRUCTION.
- ☐ (b) PROPER ARRANGEMENT HAS BEEN MADE FOR FINANCING (mortgage commitments, grants, etc.).
- ☐ (c) CONTRACTS FOR ENGINEERING SERVICES HAVE BEEN EXECUTED.
- ☐ (d) DESIGN PLANS AND SPECIFICATIONS HAVE BEEN COMPLETED.
- ☐ (e) CONSTRUCTION HAS COMMENCED.
- ☐ (f) CONSTRUCTION AND/OR EQUIPMENT ACQUISITION IS ON SCHEDULE.
- ☐ (g) CONSTRUCTION HAS BEEN COMPLETED.
- ☐ (h) START-UP HAS COMMENCED.
- ☐ (i) THE PERMITTEE HAS REQUESTED AN EXTENSION OF TIME.

SECTION K - Self-Monitoring Program

Part 1 - Flow measurement (Further explanation attached _____)

PERMITTEE FLOW MEASUREMENT MEETS THE REQUIREMENTS AND INTENT OF THE PERMIT.

☒ YES☐ NO☐ N/A

DETAILS: ULTRASONIC DETECTOR

(a) PRIMARY MEASURING DEVICE PROPERLY INSTALLED.

☒ YES☐ NO☐ N/ATYPE OF DEVICE: ☒ WEIR ☐ PARSHALL FLUME ☐ MAGMETER ☐ VENTURIMETER ☐ OTHER (Specify: 003 IMPELLER)

(b) CALIBRATION FREQUENCY ADEQUATE. (Date of last calibration: 1/18/00) 3/18/00

☒ YES☐ NO☐ N/A

(c) PRIMARY FLOW MEASURING DEVICE PROPERLY OPERATED AND MAINTAINED.

☒ YES☐ NO☐ N/A

(d) SECONDARY INSTRUMENTS (totalizers, recorders, etc.) PROPERLY OPERATED AND MAINTAINED.

☒ YES☐ NO☐ N/A

(e) FLOW MEASUREMENT EQUIPMENT ADEQUATE TO HANDLE EXPECTED RANGES OF FLOW RATES.

☒ YES☐ NO☐ N/A

Part 2 - Sampling (Further explanation attached _____)

PERMITTEE SAMPLING MEETS THE REQUIREMENTS AND INTENT OF THE PERMIT.

☒ YES☐ NO☐ N/A

DETAILS:

(a) LOCATIONS ADEQUATE FOR REPRESENTATIVE SAMPLES.

☒ YES☐ NO☐ N/A

(b) PARAMETERS AND SAMPLING FREQUENCY AGREE WITH PERMIT.

☒ YES☐ NO☐ N/A

(c) PERMITTEE IS USING METHOD OF SAMPLE COLLECTION REQUIRED BY PERMIT.

☒ YES☐ NO☐ N/AIF NO ☐ GRAB ☐ MANUAL COMPOSITE ☐ AUTOMATIC COMPOSITE FREQUENCY

(d) SAMPLE COLLECTION PROCEDURES ARE ADEQUATE.

☒ YES☐ NO☐ N/A

(i) SAMPLES REFRIGERATED DURING COMPOSITING

☒ YES☐ NO☐ N/A

(ii) PROPER PRESERVATION TECHNIQUES USED

☒ YES☐ NO☐ N/A

(iii) FLOW PROPORTIONED SAMPLES OBTAINED WHERE REQUIRED BY PERMIT

☐ YES☒ NO☐ N/A

(iv) SAMPLE HOLDING TIMES PRIOR TO ANALYSES IN CONFORMANCE WITH 40 CFR 136.3

☐ YES☒ NO☐ N/A

(e) MONITORING AND ANALYSES BEING PERFORMED MORE FREQUENTLY THAN REQUIRED BY PERMIT.

☒ YES☐ NO☐ N/A

(f) IF (e) IS YES, RESULTS ARE REPORTED IN PERMITTEE'S SELF-MONITORING REPORT.

☒ YES☐ NO☐ N/A

Part 3 - Laboratory (Further explanation attached _____)

PERMITTEE LABORATORY PROCEDURES MEET THE REQUIREMENTS AND INTENT OF THE PERMIT.

☐ YES☐ NO☐ N/A

DETAILS: WATER PARAMETERS ARE ANALYSED @ VICORAMA FACILITY OTHER THAN PH & TSS

(a) EPA APPROVED ANALYTICAL TESTING PROCEDURES USED. (40 CFR 136.3)

☒ YES☐ NO☐ N/A

(b) IF ALTERNATE ANALYTICAL PROCEDURES ARE USED, PROPER APPROVAL HAS BEEN OBTAINED.

☐ YES☐ NO☒ N/A

(c) PARAMETERS OTHER THAN THOSE REQUIRED BY THE PERMIT ARE ANALYZED.

☐ YES☒ NO☐ N/A

(d) SATISFACTORY CALIBRATION AND MAINTENANCE OF INSTRUMENTS AND EQUIPMENT.

☒ YES☐ NO☐ N/A

(e) QUALITY CONTROL PROCEDURES USED. TSS COMPARED AGAINST STANDARDS

☒ YES☐ NO☐ N/A

(f) DUPLICATE SAMPLES ARE ANALYZED. _____ % OF TIME.

☐ YES☐ NO☐ N/A

(g) SPIKED SAMPLES ARE USED. _____ % OF TIME.

☐ YES☐ NO☐ N/A

(h) COMMERCIAL LABORATORY USED. WET TEST ONLY

☒ YES☐ NO☐ N/A

(i) COMMERCIAL LABORATORY STATE CERTIFIED.

☐ YES☐ NO☒ N/A

LAB NAME COMMONWEALTH TECHNOLOGIES

LAB ADDRESS LEXINGTON KENTUCKY

PERMIT NO.

MO 0000281

SECTION L - Effluent/Receiving Water Observations (Further explanation attached _____)

| OUTFALL NO. | OIL SHEEN | GREASE | TURBIDITY | VISIBLE FOAM | VISIBLE FLOAT SOL | COLOR | OTHER |
|-------------|--------------|--------|-----------|--------------|-------------------|-------|-------|
| 001 | NO | NO | NO | NO | NO | CLEAR | — |
| 002 | NO DISCHARGE | | | | | | |
| 003 | NO | NO | NO | NO | NO | CLEAR | — |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

(Sections M and N: Complete as appropriate for sampling inspections)

SECTION M - Sampling Inspection Procedures and Observations (Further explanation attached _____)

- ☒ GRAB SAMPLES OBTAINED
☒ COMPOSITE OBTAINED
☐ FLOW PROPORTIONED SAMPLE
☒ AUTOMATIC SAMPLER USED
☒ SAMPLE SPLIT WITH PERMITTEE
☒ CHAIN OF CUSTODY EMPLOYED
☐ SAMPLE OBTAINED FROM FACILITY SAMPLING DEVICE

FLOW FOR 2/22/99 OUTFALL 001
438,620 GAL/DAY

COMPOSITING FREQUENCY EVERY 1/2 HOUR - 724 ML PRESERVATION MEALS - HNO₃, AMMONIA H₂SO₄SAMPLE REFRIGERATED DURING COMPOSITING: ☒ YES ☐ NOSAMPLE REPRESENTATIVE OF VOLUME AND NATURE OF DISCHARGE YES

SECTION N - Analytical Results (Attach report if necessary)

Acute Toxicity Test

on

Doe Run Smelter

Herculaneum, Missouri

NPDES Permit Number: MO-0000281

Activity WLR46 Sample 102

22 February to 23 February 1999

performed by

Region VII Bioassay Laboratory

Michael W. Tucker
Paul L. Andrews

APPROVED: Michael W. Tucker Date 8 March 1999
Team Leader, Bioassay Laboratory
RB- 3/11/99

ABSTRACT

Acute toxicity was observed for the Doe Run Smelter effluent collected 22 February to 23 February 1999 (activity WLR46, sample 102). Forty-eight hour static acute toxicity tests using the invertebrate Ceriodaphnia dubia, and the fathead minnow, Pimephales promelas, were performed at the U.S. EPA Region VII Bioassay Laboratory in Kansas City. Each species was exposed to 100% effluent and four lower percentage effluent concentrations containing 50.0, 25.0, 12.5, and 6.25% effluent volumes. All thirty P. promelas exposed to the 100% effluent, fifteen out of thirty in the 50.0%, and eight out of thirty in the 25.0% effluent (all statistically significant) died by the end of the 48-hour test. There were no significant mortalities observed in the two lowest percentage effluent test concentrations. Significant mortality was also observed for C. dubia, as eight out of the twenty in the 100% effluent were dead after 48 hours of exposure. No mortality was observed in any of the four lower percentage effluent test concentrations or the controls. The 48-hour LC50 value for P. promelas exposed to the Doe Run Smelter effluent was calculated to occur at 39.7% effluent. The 48-hour LC50 for C. dubia was not able to be calculated, as less than 50% mortality was observed.

I. INTRODUCTION

The purpose of this test was to determine the acute toxicity of effluent from the Doe Run Smelter, located in Herculaneum, Missouri (activity WLR46, sample 102). Test organisms used were the freshwater invertebrate, Ceriodaphnia dubia, and the fathead minnow, Pimephales promelas. Definitive acute toxicity tests were conducted from 22 February to 23 February 1999 in the Region VII Bioassay Laboratory, Kansas City, Kansas.

II. TEST ORGANISMS

A. C. dubia - The genetic stock of C. dubia used in this acute toxicity test were originally obtained from the U.S. EPA, Cincinnati, Ohio. C. dubia are cultured in the Region VII Laboratory. For information on cultivation of C. dubia refer to Region VII Bioassay Laboratory SOP (Standard Operating Procedure) "Culturing Ceriodaphnia dubia."

B. P. promelas - The fathead minnows used in this acute toxicity test were received as larvae from the U.S. EPA, Cincinnati, Ohio. Fish are maintained in the Region VII Bioassay Laboratory until used for acute toxicity tests between the ages of 1 and 14 days. Prior to test initiation, fish were acclimated to the laboratory reconstituted hard water for several hours. For further information on care of P. promelas refer to Region VII Bioassay Laboratory SOP "Hatching and Care of Fathead Minnows (Pimephales promelas)."

III. MATERIALS & METHODS

A. Procedures used in the acute toxicity tests are described in U.S. EPA Region VII Standard Operating Procedures (SOPs): "Static Acute Toxicity Test: Ceriodaphnia dubia" and "Static Acute Toxicity Test: Pimephales promelas (Fathead Minnow)." These adhere to the guidelines in Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (U.S. EPA, 1993).

B. The effluent tested was collected by Bruce Littell of the U.S. EPA Region VII Environmental Monitoring and Water Compliance Branch. A 24-hour composite sample of effluent was collected 22 February to 23 February 1999 and was received in the Region VII Bioassay Laboratory on 24 February 1999. The toxicity test was initiated on the day of sample receipt.

C. Testing was performed using concentrations of 100, 50.0, 25.0, 12.5, and 6.25% effluent. Controls, consisting of 100% dilution water, were also used. The dilution water was reconstituted hard water (hardness = 220 mg/L CaCO₃) prepared by Region VII personnel.

D. C. dubia Acute Methods - This static toxicity test was conducted using 30 ml polystyrene cups as test chambers. Each chamber was given 20 ml of test solution which was not renewed during the 48-hour test period. There were four chambers for each effluent concentration and four with control water. At the start of the test, food was added to each chamber equivalent to the daily amount given during culture maintenance. Five C. dubia neonates, ≤ 24 hours old, were randomly selected and added to each test chamber. A total of 20 organisms per concentration were tested. Observations for mortality were made after 24 and 48 hours of exposure.

E. P. promelas Acute Methods - This static toxicity test was conducted using 500 ml polypropylene cups as test chambers. For each effluent concentration and control water, 400 ml aliquots were dispensed into each of three test chambers. Test solutions were not renewed during the 48-hour test period. Ten P. promelas, 5-days old, were randomly selected and placed in each test chamber. A total of 30 organisms per concentration were tested. Fish were not fed during the test. Observations for mortality were made after 24 and 48 hours of exposure.

F. Water Quality Methods - Prior to test initiation, temperature, pH, dissolved oxygen (DO), conductivity, alkalinity, hardness, and total residual chlorine were measured for the sample effluent that was received in the Bioassay Laboratory. These parameters were also measured for the control water. At 24 hours, temperature and DO were measured in fish chambers for each test concentration to verify that DO levels remained above 4.0 mg/L. At test termination, temperature, dissolved oxygen, pH, and conductivity were measured on controls and each test concentration for both species.

G. Data Analysis - Statistically significant ($p \leq 0.05$) death was assessed by either Dunnett's Procedure (P. promelas) or Steel's Many-One Rank Test (C. dubia), using average percent survival of each test concentration versus the average survival for controls. When $\geq 50\%$ mortality occurs, effluent test concentrations and corresponding percent mortality data are used to estimate the 24-hour and 48-hour median lethal effect concentrations (LC50) and 95% confidence intervals where appropriate by the Trimmed Spearman-Kärber method.

IV. RESULTS

A. C. dubia Mortality Results - Effluent collected at the Doe Run Smelter exhibited acute toxicity to the freshwater invertebrate, C. dubia. Statistically significant mortality was observed, as eight out of the twenty C. dubia exposed to the 100% effluent died by the end of the 48-hour test. There were no mortalities in any of the four lower percentage effluent test concentrations or the controls (Table 1). The 48-hour LC50 for C. dubia was not able to be calculated, as less than 50% mortality was observed in the 100% effluent.

Table 1. C. dubia Cumulative Mortality Results.

| Effluent Concentration (%) | Organisms Exposed | 24-Hour Mortality | 48-Hour Mortality | 48-Hour Mortality (%) |
|----------------------------|-------------------|-------------------|-------------------|-----------------------|
| * 100 | 20 | 4 | *8 | *40 |
| 50.0 | 20 | 0 | 0 | 0 |
| 25.0 | 20 | 0 | 0 | 0 |
| 12.5 | 20 | 0 | 0 | 0 |
| 6.25 | 20 | 0 | 0 | 0 |
| Control | 20 | 0 | 0 | 0 |

* = The average percent survival was significantly less than control organism survival ($p \leq 0.05$, Steel's Test).

B. P. promelas Mortality Results - Effluent collected at the Doe Run Smelter exhibited acute toxicity to the fathead minnow, P. promelas. Thirty out of the thirty 5-day-old P. promelas exposed to the 100% effluent died prior to test termination. Statistically significant mortality was also observed in test concentrations containing 50.0% effluent (15 out of 30 died) and 25.0% effluent (8 out of 30 died.) Significant mortality was not observed in the 12.5% effluent, and no mortality was observed in the 6.25% effluent and in the controls. The 48-hour LC50 was calculated to occur at 39.7% effluent, with 95% confidence limits of 33.1% and 47.5% effluent. The 24-hour LC50 was calculated to occur at 43.5% effluent with 95% confidence limits of 36.6 to 51.8% effluent.

Table 2. P. promelas (Fathead Minnow) Cumulative Mortality.

| Effluent Concentration (%) | Organisms Exposed | 24-Hour Mortality | 48-Hour Mortality | 48-Hour Mortality (%) |
|----------------------------|-------------------|-------------------|-------------------|-----------------------|
| * 100 | 30 | *30 | *30 | *100 |
| * 50.0 | 30 | *13 | *15 | * 50.0 |
| * 25.0 | 30 | * 6 | * 8 | * 26.6 |
| 12.5 | 30 | 2 | 2 | 6.7 |
| 6.25 | 30 | 0 | 0 | 0 |
| Control | 30 | 0 | 0 | 0 |

* = The average percent survival was significantly less than control organism survival ($p \leq 0.05$, Dunnett's Test).

C. Water Chemistry Results - These are presented in Tables 3, 4, and 5 and are summarized as follows:

Total residual chlorine (Cl_2): The effluent had no detectable level of total residual chlorine (< 0.02 mg/L as Cl) upon receipt in the Bioassay Laboratory.

Dissolved oxygen (DO): Dissolved oxygen was 7.6 mg/L in the 100% effluent after raising the sample to the desired test initiation temperature of 25 ± 1 degree Celsius. DO measurements taken at 24 hours did not decline lower than 7.5 mg/L in P. promelas test chambers. All fish chambers had DO levels ≥ 7.6 mg/L when measured at 48 hours. In C. dubia test chambers, DO was measured at 8.2 to 8.4 mg/L at test termination.

pH: The pH of the 100% effluent was 9.43 upon receipt in the laboratory at 6.2 degrees Celsius. At test initiation the 100% effluent pH was 9.11 at 24.6 degrees Celsius. Dilution water used was at a pH of 8.47. At test termination, pH measurements in test chambers ranged from 8.06 to 8.52 for P. promelas and from 7.86 to 8.58 for C. dubia.

Alkalinity and hardness: The effluent sample had an alkalinity of 86 mg/L and hardness of >1000 mg/L (as CaCO_3). The laboratory dilution water had an alkalinity of 148 mg/L and hardness of 220 mg/L.

Conductivity: The conductivity of the 100% effluent was higher (2.90 mMhos/cm) than that of the control water (0.649 mMhos/cm). The conductivity of the test concentrations decreased with increasing dilution.

Temperature: The test chamber temperatures observed at 24 and 48 hours ranged from 24.3 to 25.8 degrees Celsius for P. promelas and from 24.9 to 25.5 degrees Celsius for C. dubia.

Table 3. Water chemistry measured at test initiation. The 100% and four lower percentage effluent concentrations were prepared from Doe Run Smelter effluent. The dilution and control water consisted of reconstituted hard water.

| Effluent (%) | Cl_2 (mg/L) | Dissolved Oxygen (mg/L) | pH | Conductivity (mMhos/cm) | Alkalinity (mg/L) | Hardness (mg/L) |
|--------------|----------------------|-------------------------|------|-------------------------|-------------------|-----------------|
| 100 | ND | 7.6 | 9.11 | 2.90 | 86 | >1000 |
| 50.0 | NA | 7.8 | 8.73 | 1.940 | NA | NA |
| 25.0 | NA | 8.1 | 8.57 | 1.327 | NA | NA |
| 12.5 | NA | 8.1 | 8.50 | 1.029 | NA | NA |
| 6.25 | NA | 8.1 | 8.48 | 0.816 | NA | NA |
| Control | ND | 8.3 | 8.47 | 0.649 | 148 | 220 |

ND = nondetectable (detection limits = 0.02 mg/L as Cl)

NA = not analyzed

Table 4. C. dubia 48-Hour Water Quality Measurements.

| Effluent Concentration (%) | Temperature (C) | Dissolved Oxygen (mg/L) | pH | Conductivity (mMhos/cm) |
|----------------------------------|--------------------|-------------------------------|------|----------------------------|
| 100 | 25.1 | 8.2 | 7.86 | 3.12 |
| 50.0 | 25.5 | 8.2 | 8.14 | 2.08 |
| 25.0 | 25.2 | 8.2 | 8.35 | 1.421 |
| 12.5 | 25.0 | 8.4 | 8.43 | 1.114 |
| 6.25 | 25.0 | 8.4 | 8.50 | 0.896 |
| Control | 24.9 | 8.2 | 8.58 | 0.732 |

Table 5. P. promelas 48-Hour Water Quality Measurements.

| Effluent Concentration (%) | Temperature (C) | Dissolved Oxygen (mg/L) | pH | Conductivity (mMhos/cm) |
|----------------------------------|--------------------|-------------------------------|------|----------------------------|
| a 100 | 25.2 | 7.7 | 8.47 | 2.95 |
| 50.0 | 25.0 | 8.0 | 8.06 | 1.99 |
| 25.0 | 25.5 | 7.6 | 8.13 | 1.364 |
| 12.5 | 25.7 | 8.1 | 8.30 | 1.057 |
| 6.25 | 25.5 | 8.2 | 8.46 | 0.859 |
| Control | 25.8 | 8.4 | 8.52 | 0.677 |

a = measured at 24 hours due to complete mortality.

V. QUALITY ASSURANCE

The absence of control mortality during this test indicated the health of the test organisms and also showed that any significant mortality in test concentrations was not due to contaminants or variations in test conditions.

Reference acute toxicity tests are routinely performed by the Region VII Bioassay Laboratory.

VI. REFERENCES

Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, 4th Edition, Weber, C.I., 1993. U.S. EPA/600/4-90/027F, 293pp.

Standard Operating Procedure for Static Acute Toxicity Test: Pimephales promelas (Fathead Minnow), U.S. EPA Region VII.

Standard Operating Procedure for Static Acute Toxicity Test: Ceriodaphnia dubia, U.S. EPA Region VII.

Standard Operating Procedure for Hatching and Care of Fathead Minnows (Pimephales promelas), U.S. EPA Region VII.

Standard Operating Procedure for Culturing Ceriodaphnia dubia, U.S. EPA Region VII.

ATTACHMENT 7

STATE OF MISSOURI
DEPARTMENT OF NATURAL RESOURCES
MISSOURI CLEAN WATER COMMISSION



MISSOURI STATE OPERATING PERMIT

In compliance with the Missouri Clean Water Law, (Chapter 644 R.S. Mo. as amended, hereinafter, the Law), and the Federal Water Pollution Control Act (Public Law 92-500, 92nd Congress) as amended,

Permit No. MO-0000281

Owner: The Doe Run Company

Owner's Address: 1801 Park 270 Drive, Suite 200, St. Louis, Missouri 63146

Operating Authority: Doe Run Company Smelting Division

Operating Authority's Address: 881 Main St., Herculaneum, MO 63048

Facility Name: Doe Run - Herculaneum Smelter

Facility Address: 881 Main Street, Herculaneum, Missouri 63048

Legal Description: NE ¼, SE ¼, U.S. Survey 3028, T41N, R6E, Jefferson County

Receiving Stream & Basin: Mississippi River (Basin 48) (07140101-04-01) (P)

is authorized to discharge from the facility described herein, in accordance with the effluent limitations and monitoring requirements as set forth herein:

FACILITY DESCRIPTION

See Page Two

This permit authorizes only wastewater discharges under the Missouri Clean Water Law and the National Pollutant Discharge Elimination System; it does not apply to other regulated areas. This permit may be appealed in accordance with Section 644.051.6 of the Law.

November 9, 1995

Effective Date

March 22, 2000

Expiration Date

MO 780-0041 (10-93)

John A. Young
John A. Young
Director, Division of Environmental Quality

ORIGINAL SIGNED BY DIRECTOR OF
STAFF, CLEAN WATER COMMISSION

Director of Staff, Clean Water Commission

FACILITY DESCRIPTION - Industry - SIC #3332

Outfall #001 - Industrial process wastewater and process stormwater is treated in a wastewater plant with a design capacity of 1,152,000 gallons per day. The treatment consists of the following unit processes:

1. Flocculation
2. Neutralization
3. Sedimentation
4. Sand/Anthracite Filtration
5. Clarification
6. Sludge thickening/dewatering

Actual flow is 850 GPM.

Outfall #002 - Emergency stormwater overflow, no treatment. Design flow is 0.432 MGD.
Actual flow is 300 GPM.

Outfall #003 - Acid plant non-contact cooling water, no treatment/Non-contact cooling water. Design flow is 2.33 MGD.

The sanitary waste from the toilets is treated in the Herculaneum POTW.

A. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTSPAGE NUMBER 3 of 8
PERMIT NUMBER MO-0000281

The permittee is authorized to discharge from outfall(s) with serial number(s) as specified in the application for this permit. The final effluent limitations shall become effective upon issuance and remain in effect until expiration of the permit. Such discharges shall be controlled, limited, and monitored by the permittee as specified below:

| OUTFALL NUMBER AND EFFLUENT PARAMETER(S) | UNITS | FINAL EFFLUENT LIMITATIONS | | | MONITORING REQUIREMENTS | |
|--|------------|--|-------------------|--------------------|---------------------------------|---------------------|
| | | DAILY MAXIMUM | WEEKLY AVERAGE | MONTHLY AVERAGE | MEASUREMENT FREQUENCY | SAMPLE TYPE |
| <u>Outfall #001</u> | | | | | | |
| Flow | MGD | * | | * | once/day | 24 hr. total |
| pH - Units | SU | ** | | ** | once/month | grab |
| Total Suspended Solids | lbs/day | 170.325 | | 136.257 | once/month | grab |
| Arsenic, Total Recoverable | lbs/day | 12.861 | | 5.216 | once/month | grab |
| Cadmium, Total Recoverable | lbs/day | 1.964 | | 0.785 | once/month | grab |
| Cadmium, Total Recoverable | mg/L | 0.720 | | | once/month | grab |
| Copper, Total Recoverable | lbs/day | 11.839 | | 4.750 | once/month | grab |
| Copper, Total Recoverable | mg/L | 0.580 | | | once/month | grab |
| Lead, Total Recoverable | lbs/day | 2.749 | | 1.127 | once/month | grab |
| Lead, Total Recoverable | mg/L | 1.900 | | | once/month | grab |
| Zinc, Total Recoverable | lbs/day | 10.016 | | 3.307 | once/month | grab |
| Zinc, Total | mg/L | 4.900 | | | once/month | grab |
| MONITORING REPORTS | SHALL BE | SUBMITTED <u>MONTHLY</u> , THE FIRST REPORT | | | IS DUE <u>December 28, 1995</u> | |
| Silver | mg/L | 0.130 | | | once/year in September | grab |
| Whole Effluent Toxicity (WET) Test | % Survival | (See Special Conditions) | | | once/year | 24 hr. composite |
| MONITORING REPORTS | SHALL BE | SUBMITTED <u>ANNUALLY</u> , THE FIRST REPORT | | | IS DUE <u>October 28, 1996</u> | |
| | | | | | | |

MONITORING REPORTS SHALL BE SUBMITTED as outlined above; THE FIRST REPORT IS DUE as outlined above
THERE SHALL BE NO DISCHARGE OF FLOATING SOLIDS OR VISIBLE FOAM IN OTHER THAN TRACE AMOUNTS.

B. STANDARD CONDITIONS

IN ADDITION TO SPECIFIED CONDITIONS STATED HEREIN, THIS PERMIT IS SUBJECT TO THE ATTACHED Part I
STANDARD CONDITIONS DATED October 1, 1980, AND HEREBY INCORPORATED AS THOUGH FULLY SET FORTH HEREIN.

A. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS (continued)

PAGE NUMBER 4 of 8
PERMIT NUMBER MO-0000281

| OUTFALL NUMBER AND EFFLUENT PARAMETER(S) | UNITS | FINAL EFFLUENT LIMITATIONS | | | MONITORING REQUIREMENTS | |
|---|------------|----------------------------|----------------|-----------------|-----------------------------|-------------|
| | | DAILY MAXIMUM | WEEKLY AVERAGE | MONTHLY AVERAGE | MEASUREMENT FREQUENCY | SAMPLE TYPE |
| <u>Outfall #002</u> | | | | | | |
| Flow | MGD | * | | * | once/weekday***24 hr. total | |
| pH - Units | SU | ** | | ** | (Note 1) | grab |
| Total Suspended Solids | lbs/day | (Note 1) | | | (Note 1) | grab |
| Arsenic, Total Recoverable | lbs/day | (Note 1) | | | (Note 1) | grab |
| Cadmium, Total Recoverable | lbs/day | (Note 1) | | | (Note 1) | grab |
| Copper, Total Recoverable | lbs/day | (Note 1) | | | (Note 1) | grab |
| Lead, Total Recoverable | lbs/day | (Note 1) | | | (Note 1) | grab |
| Zinc, Total Recoverable | lbs/day | (Note 1) | | | (Note 1) | grab |
| Whole Effluent Toxicity (WET) Test | † Survival | (See Note 2) | | | once/year | grab |
| MONITORING REPORTS SHALL BE SUBMITTED <u>ANNUALLY</u> , THE FIRST REPORT IS DUE <u>October 28, 1996</u> | | | | | | |
| <u>Outfall #003</u> | | | | | | |
| Flow | MGD | * | | | once/weekday***24 hr. total | |
| Temperature | °F | * | | | once/weekday***grab | |
| pH | SU | **** | | | once/week | grab |
| Arsenic, Total Recoverable | mg/L | * | | | once/month | grab |
| Cadmium, Total Recoverable | mg/L | * | | | once/month | grab |
| Copper, Total Recoverable | mg/L | * | | | once/month | grab |
| Lead, Total Recoverable | mg/L | * | | | once/month | grab |
| Zinc, Total Recoverable | mg/L | * | | | once/month | grab |
| MONITORING REPORTS SHALL BE SUBMITTED <u>MONTHLY</u> , THE FIRST REPORT IS DUE <u>December 28, 1995</u> | | | | | | |
| | | | | | | |

A. EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS (continued)

PAGE NUMBER 5 of 8
PERMIT NUMBER MO-0000281

| OUTFALL NUMBER AND EFFLUENT PARAMETER(S) | UNITS | FINAL EFFLUENT LIMITATIONS | | | MONITORING REQUIREMENTS | |
|---|-------|----------------------------|-------------------|--------------------|--------------------------|----------------|
| | | DAILY MAXIMUM | WEEKLY AVERAGE | MONTHLY AVERAGE | MEASUREMENT FREQUENCY | SAMPLE TYPE |
| <p>* Monitoring requirement only.</p> <p>** pH is measured in pH units and is not to be averaged. The pH is limited to the range of 7.5-10.0 pH units.</p> <p>*** Once each weekday means: Monday, Tuesday, Wednesday, Thursday and Friday.</p> <p>**** pH is measured in pH units and is not to be averaged. The pH is limited to the range of 6.0-9.0 pH units.</p> <p>Note 1 - Once per month during any month in which a discharge occurs from Outfall #002, the discharge from both Outfalls #001 and #002 shall be sampled on the same day and reported together. The combined effluents discharging from both Outfalls #001 and #002 shall not exceed the discharge mass limitations set for Outfall #001. If no discharge occurs from Outfall #002, then report no discharge from Outfall #002.</p> <p>Note 2 - Once per year during a month in which a discharge occurs from Outfall #002, a sample shall be collected and a WET test run in conformance with special condition number 7. If no discharge occurs from Outfall #002 during the year, then report no discharge from Outfall #002 during the past year.</p> | | | | | | |

C. SCHEDULE OF COMPLIANCE

1. By November 9, 1996, the Doe Run Company shall provide the Department of Natural Resources with the results of a water tightness pressure test of the emergency discharge pipe between the sump and the vertical terminus of the pipe. Also include a copy of standard operational procedures which are used when a sump pump failure occurs, showing expected response time to effect repairs. If the results show that the rate of water loss is within the design guidelines contained in 10 CSR 20-8.120(6)(H)2 for the size and length of this pipe, no further action is required. If the results show a rate of loss greater than allowed in this regulation, this permit may be modified, or alternatively revoked and reissued, to incorporate new or modified effluent monitoring, limitations or other conditions.

D. SPECIAL CONDITIONS

- 1) Any sludge removed shall be processed through the smelting process, or the Missouri Department of Natural Resources shall be contacted for approval of the alternate disposal proposal.
 - 2) The permittee shall notify the Director as soon as they know or have reason to believe:
 - (A) That any activity has occurred or will occur which would result in the discharge of any toxic pollutant which is not limited in the permit, if that discharge will exceed the highest of the following "notification levels":
 - 1) One hundred micrograms per liter (100 µg/l);
 - 2) Two hundred micrograms per liter (200 µg/l) for acrolein and acrylonitrile; five hundred micrograms per liter (500 µg/l) for 2,4 dinitrophenol and for 2-methyl-4, 6-dinitrophenol; and one milligram per liter (1 mg/l) for antimony;
 - 3) Five (5) times the maximum concentration value reported for the pollutant in the permit application;
 - 4) The level established in Part A of the permit by the Director.
 - (B) That they have begun or expect to begin to use or manufacture as an intermediate or final product or byproduct any toxic pollutant which was not reported in the permit application.
 - 3) This permit may be modified, or alternatively revoked and reissued, to comply with any applicable effluent standard or limitation issued or approved under Sections 301(b)(2)(C) and (D), 304(b)(2) and 307(a)(2) of the Clean Water Act, if the effluent standard or limitation so issued or approved:
 - (A) Contains different conditions or is otherwise more stringent than any effluent limitation in the permit; or
 - (B) Controls any pollutant not limited in the permit.
- The permit as modified or reissued under this paragraph shall also contain any other requirements of the Act then applicable.
- 4) This permit may be modified, or alternatively revoked and reissued, to incorporate new or modified effluent limitations or other conditions, if the results of a wasteload allocation study, toxicity test, or other information indicates changes are necessary to assure compliance with Missouri's Water Quality Standards.
 - 5) Discharges shall not cause violations of the general criteria as defined in the Water Quality Standards 10 CSR 20-7.031 (3) including but not limited to the following criteria:
 - (A) Waters shall be free from substances in sufficient amounts to cause formation of putrescents, unsightly or harmful bottom deposits or prevent full maintenance of beneficial uses;
 - (B) Waters shall be free from oil, scum and floating debris in sufficient amounts to be unsightly or prevent full maintenance of beneficial uses;
 - (C) Waters shall be free from substances in sufficient amounts to cause unsightly color or turbidity, offensive odor or prevent full maintenance of beneficial uses; and
 - (D) Waters shall be free from substances or conditions in sufficient amounts to have a harmful effect on human, animal or aquatic life.
 - 6) A Quality assurance/Quality control (QA/QC) plan shall be maintained for samples analyzed by the permittee, and QA/QC plans submitted for any other laboratories which will be used to fulfill monitoring requirements.

D. SPECIAL CONDITIONS (continued)

7. Whole Effluent Toxicity (WET) tests will be conducted as follows:

| SUMMARY OF WET TESTING FOR THIS PERMIT | | | | |
|--|----------|-----------|--------------|----------|
| OUTFALL | A.E.C. % | FREQUENCY | SAMPLE TYPE | MONTH |
| Outfalls 001-002 | 10% | Annually | 24 hr. comp. | December |

a. Test Schedule and Follow-Up Requirements

- (1) Perform a single-dilution test in the months and at the frequency specified above.

If the test passes the effluent limit do not repeat test until the next test period. Submit results with the annual report.

If the test fails the effluent limit a multiple dilution test shall be performed within 30 days, and biweekly thereafter until one of the following conditions are met:

(a) THREE CONSECUTIVE MULTIPLE-DILUTION TESTS PASS. No further tests need to be performed until next regularly scheduled test period.

(b) A TOTAL OF THREE MULTIPLE-DILUTION TESTS FAIL.

- (2) The permittee shall submit a summary of all test results for the test series to the Planning Section of the WPCP, DNR, Box 176, Jefferson City, MO within 14 days of the third failed test. DNR will contact the permittee with initial guidance on conducting a toxicity identification evaluation (TIE) or toxicity reduction evaluation (TRE). The permittee shall submit a plan for conducting a TIE or TRE to the Planning Section of the WPCP within 60 days of the date of DNR's letter. This plan must be approved by DNR before the TIE or TRE is begun. A schedule for completing the TIE or TRE shall be established in the plan approval.
- (3) Upon DNR's approval, the TIE/TRE schedule may be modified if toxicity is intermittent during the TIE/TRE investigations. A revised WET test schedule may be established by DNR for this period.
- (4) If a previously completed TIE has clearly identified the cause of toxicity, additional TIEs will not be required as long as effluent characteristics remain essentially unchanged and the permittee is proceeding according to a DNR approved schedule to complete a TRE and reduce toxicity. Regularly scheduled WET testing as required in part b. (1) will be required during this period.
- (5) In addition to the WET test summary report required in part (5), all failing test results shall be reported to DNR within 14 days of the availability of results.
- (6) All WET test results for the reporting period shall be summarized and submitted to DNR by the end of the following October. When WET test sampling is required to run over one DMR period, each DMR report shall contain information generated during the reporting period.

b. PASS/FAIL procedure and effluent limitations

- (1) To pass a single-dilution test, mortality observed in the AEC test concentration shall not be significantly different (at the 95% confidence level; $p = 0.05$) than that observed in the upstream receiving-water control. The appropriate statistical tests of significance will be those outlined in the most current USEPA acute toxicity manual or those specified by the MDNR.

(continued)

D. SPECIAL CONDITIONS (continued)

Whole Effluent Toxicity (WET) Test (continued)

- (2) To pass a multiple-dilution test:
 - (a) the computed percent effluent at the edge of the zone of initial dilution (AEC) must be less than three-tenths (0.3) of the LC_{50} concentration for the most sensitive of the test organisms, or,
 - (b) all dilutions equal to or greater than the AEC must be nontoxic. Failure of one multiple-dilution test is considered an effluent limit violation.

c. Test Conditions

- (1) Test species: Ceriodaphnia dubia and fathead minnows, Pimephales promelas. Organisms used in WET testing should come from cultures reared for the purpose of conducting toxicity tests and should be cultured in a manner consistent with the most current USEPA guidelines. All test animals should be cultured as described in EPA-600/4-90/027.
- (2) Test period: 48 hours at the "Acceptable Effluent Concentration" (AEC) specified above.
- (3) When dilutions are required, upstream receiving stream water will be used as dilution water. If upstream water is unavailable or if mortality in the upstream water exceeds 10%, "reconstituted" water will be used. Procedures for generating reconstituted water will be supplied by the Department of Natural Resources (DNR).
- (4) Tests should be initiated immediately after the sample is collected, but tests must be initiated no later than 36 hours after collection.
- (5) Single-dilution tests will be run with:
 - (a) Effluent at the AEC concentration;
 - (b) 100% receiving-stream water (if available), collected upstream of the outfall at a point beyond any influence of the effluent; and
 - (c) reconstituted water.
- (6) Multiple-dilution tests will be run with:
 - (a) 100%, 50%, 25%, 12.5%, and 6.25% effluent, unless the AEC is less than 25% effluent, in which case dilutions will be 4 times the AEC, two times the AEC, AEC, 1/2 AEC and 1/4 AEC.
 - (b) 100% receiving-stream water (if available), collected upstream of the outfall at a point beyond any influence of the effluent; and
 - (c) reconstituted water.
- (7) If reconstituted-water control mortality for a test species exceeds 10%, the entire test will be rerun.

ATTACHMENT 5

ANALYSIS REQUEST REPORT

VALIDATED DATA

FOR ACTIVITY: WLR46

LITTELL, BRUCE

04/16/99 11:19:40

ALL REAL SAMPLES AND FIELD Q.C.

* FINAL REPORT

FY: 99 ACTIVITY: WLR46 DESCRIPTION: DOE RUN-HERCULANEUM SMELTER LOCATION: HERCULANEUM MISSOURI
 STATUS: ACTIVE TYPE: SAMPLING - IN HOUSE ANALYSIS PROJECT: W43
 LABO DUE DATE IS 3/26/99. REPORT DUE DATE IS 4/15/99.
 INSPECTION DATE: 2/23/99 ALL SAMPLES RECEIVED DATE: 02/24/99
 ALL DATA APPROVED BY LABO DATE: 03/10/99 FINAL REPORT TRANSMITTED DATE: 04/20/99
 EXPECTED LABO TURNAROUND TIME IS 30 DAYS EXPECTED REPORT TURNAROUND TIME IS 51 DAYS
 ACTUAL LABO TURNAROUND TIME IS 14 DAYS ACTUAL REPORT TURNAROUND TIME IS 56 DAYS
 SITE CODE: SITE:

| SAMP. NO. | QCC | M | DESCRIPTION | SAMPLE # STATUS | CITY | STATE | AIRS/ STORET LOC NO | LAY- SECT ER | BEG. DATE | BEG. TIME | END. DATE | END. TIME |
|--------------|-----|---|------------------------------------|--------------------|-------------|----------|---------------------------|-----------------|--------------|--------------|--------------|--------------|
| 001 | F | W | FIELD BLANK | 1 | HERCULANEUM | MISSOURI | | | 02/23/99 | 13:15 | / / | : |
| 101 | | W | DOE RUN-HERCULANEUM SMELT. OUTFALL | 001 1 | HERCULANEUM | MISSOURI | | | 02/23/99 | 13:15 | / / | : |
| 102 | | W | DOE RUN-HERCULANEUM SMELT. OUTFALL | 001 1 | HERCULANEUM | MISSOURI | | | 02/22/99 | 14:45 | 02/23/99 | 13:06 |
| 301 | | W | DOE RUN-HERCULANEUM OUTFALL | 003 1 | HERCULANEUM | MISSOURI | | | 02/23/99 | 13:45 | / / | : |

EXPLANATION OF CODES AND INFORMATION ON ANALYSIS REQUEST DETAIL REPORT

SAMPLE INFORMATION:

SAMP. NO. = SAMPLE IDENTIFICATION NUMBER (A 3-DIGIT NUMBER WHICH IN COMBINATION WITH THE ACTIVITY NUMBER AND QCC, PROVIDES AN UNIQUE NUMBER FOR EACH SAMPLE FOR IDENTIFICATION PURPOSES)

QCC = QUALITY CONTROL CODE (A ONE-LETTER CODE USED TO DESIGNATE SPECIFIC QC SAMPLES. THIS FIELD WILL BE BLANK FOR ALL NON-QC OR ACTUAL SAMPLES):

B = CAL INCREASED CONCENTRATION FOR A LAB SPIKED DUP SAMPLE

D = MEASURED VALUE FOR FIELD DUPLICATE SAMPLE

F = MEASURED VALUE FOR FIELD BLANK

G = MEASURED VALUE FOR METHOD STANDARD

H = TRUE VALUE FOR METHOD STANDARD

K = CAL INCREASED CONCENTRATION FOR FIELD SPIKED DUP SAMPLE

L = MEASURED VALUE FOR A LAB DUPLICATE SAMPLE

M = MEASURED VALUE FOR LAB BLANK

N = MEASURED CONCENTRATION OF FIELD SPIKED DUPLICATE

P = MEASURED VALUE FOR PERFORMANCE STANDARD

R = CAL INCREASED CONCENTRATION RESULTING FROM LAB SPIKE

S = MEASURED CONCENTRATION OF LAB SPIKED SAMPLE

T = TRUE VALUE OF PERFORMANCE STANDARD

U = MEASURED CONCENTRATION OF LAB SPIKED DUPLICATE

Y = MEASURED CONCENTRATION OF FIELD SPIKED SAMPLE

Z = CAL INCREASED CONCENTRATION RESULTING FROM FIELD SPIKE

1 = MEASURED VALUE OF FIRST SPIKED REPLICATE

2 = MEASURED VALUE OF SECOND SPIKED REPLICATE

3 = MEASURED VALUE OF THIRD SPIKED REPLICATE

4 = MEASURED VALUE OF FOURTH SPIKED REPLICATE

5 = MEASURED VALUE OF FIFTH SPIKED REPLICATE

6 = MEASURED VALUE OF SIXTH SPIKED REPLICATE

7 = MEASURED VALUE OF SEVENTH SPIKED REPLICATE

M = MEDIA CODE (A ONE-LETTER CODE DESIGNATING THE MEDIA OF THE SAMPLE):

A = AIR H = HAZARDOUS WASTE/OTHER

S = SOLID (SOIL, SEDIMENT, SLUDGE)

T = TISSUE (PLANT & ANIMAL)

W = WATER (GROUND WATER, SURFACE WATER, WASTE WATER, DRINKING WATER)

DESCRIPTION = A SHORT DESCRIPTION OF THE LOCATION WHERE SAMPLE WAS COLLECTED

AIRS/STORET LOC. NO. = THE SPECIFIC LOCATION ID NUMBER OF EITHER OF THESE NATIONAL DATABASE SYSTEMS, AS APPROPRIATE

DATE/TIME INFORMATION = SPECIFIC INFORMATION REGARDING WHEN THE SAMPLE WAS COLLECTED

BEG. DATE = DATE SAMPLING WAS STARTED

BEG. TIME = TIME SAMPLING WAS STARTED

END DATE = DATE SAMPLING WAS COMPLETED

END TIME = TIME SAMPLING WAS COMPLETED

NOTE: A GRAB SAMPLE WILL CONTAIN ONLY BEG. DATE/TIME

A TIMED COMPOSITE SAMPLE WILL CONTAIN BOTH BEG AND END DATE/TIME TO DESIGNATE DURATION OF SAMPLE COLLECTION

OTHER CODES

V = VALIDATED

ANALYTICAL RESULTS/MEASUREMENTS INFORMATION:

COMPOUND = MGP (MEDIA-GROUP-PARAMETER) CODE AND NAME OF THE MEASURED CONSTITUENT OR CHARACTERISTIC OF EACH SAMPLE

UNITS = SPECIFIC UNITS IN WHICH RESULTS ARE REPORTED:

C = CENTIGRADE (CELSIUS) DEGREES

CFS = CUBIC FEET PER SECOND

GPM = GALLONS PER MINUTE

IN = INCHES

I.D. = SPECIES IDENTIFICATION

KG = KILOGRAM

L = LITER

LB = POUNDS

MG = MILLIGRAMS (1 X 10⁻³ GRAMS)

MGD = MILLION GALLONS PER DAY

MPH = MILES PER HOUR

MV = MILLIVOLT

M/F = MALE/FEMALE

M2 = SQUARE METER

M3 = CUBIC METER

NA = NOT APPLICABLE

NG = NANOGRAMS (1 X 10⁻⁹ GRAMS)

NTU = NEPHELOMETRIC TURBIDITY UNITS

PC/L = PICO (1 X 10⁻¹²) CURRIES PER LITER

PG = PICOGRAMS (1 X 10⁻¹² GRAMS)

P/CM2 = PICOGRAMS PER SQUARE CENTIMETER

SCH = STANDARD CUBIC METER (1 ATM, 25 C)

SQ FT = SQUARE FEET

SU = STANDARD UNITS (PH)

UG = MICROGRAMS (1 X 10⁻⁶ GRAMS)

UNHOS = MICROMHOS/CM (CONDUCTIVITY UNITS)

U/CC2 = MICROGRAMS PER 100 SQUARE CENTIMETERS

U/CM2 = MICROGRAMS PER SQUARE CENTIMETER

1000G = 1000 GALLONS

+/- = POSITIVE/NEGATIVE

= NUMBER

DATA QUALIFIERS = SPECIFIC CODES USED IN CONJUNCTION WITH DATA VALUES TO PROVIDE ADDITIONAL INFORMATION ON THE REPORTED RESULTS, OR USED TO EXPLAIN THE ABSENCE OF A SPECIFIC VALUE:

BLANK = IF FIELD IS BLANK, NO REMARKS OR QUALIFIERS ARE PERTINENT. FOR FINAL REPORTED DATA, THIS MEANS THAT THE VALUES HAVE BEEN REVIEWED AND FOUND TO BE ACCEPTABLE FOR USE.

I = INVALID SAMPLE/DATA - VALUE NOT REPORTED

J = THE ASSOCIATED NUMERICAL VALUE IS AN ESTIMATED QUANTITY

K = ACTUAL VALUE OF SAMPLE IS < VALUE REPORTED

L = ACTUAL VALUE OF SAMPLE IS > VALUE REPORTED

M = DETECTED BUT BELOW THE LEVEL OF REPORTED VALUE FOR ACCURATE QUANTIFICATION

O = PARAMETER NOT ANALYZED

U = THE MATERIAL WAS ANALYZED FOR, BUT WAS NOT DETECTED. THE ASSOCIATED NUMERICAL VALUE IS THE SAMPLE DETECTION LIMIT.

ANALYSIS REQUEST DETAIL REPORT

ACTIVITY: 9-WLR46

VALIDATED DATA

| COMPOUND | UNITS | 001 | F | 101 | 102 | 301 |
|---|-------|-------|----|-------|-----|----------|
| WB27 TOXICITY, ACUTE, FISH | +/- | | | | POS | |
| WB29 TOXICITY, ACUTE, CERIODAPHNIA | +/- | | | | POS | |
| WF01 TEMPERATURE, WATER | 'C | | | 32.3 | V | |
| WF05 PH, FIELD | SU | | | 9.15 | V | 7.02 V |
| WG24 SOLIDS, TOTAL NON-FILTERABLE (NFS) | MG/L | | | 15.0 | V | |
| WM01 SILVER, TOTAL, BY ICAP | UG/L | 7.24 | UV | 7.24 | UV | 7.24 UV |
| WM02 ALUMINUM, TOTAL, BY ICAP | UG/L | 33.5 | UV | 88.7 | V | 33.5 UV |
| WM03 ARSENIC, TOTAL, BY ICAP | UG/L | 7.14 | UV | 7.14 | UV | 7.14 UV |
| WM04 BARIUM, TOTAL, BY ICAP | UG/L | 0.743 | UV | 113 | V | 86.5 V |
| WM05 BERYLLIUM, TOTAL, BY ICAP | UG/L | 0.420 | UV | 0.420 | UV | 0.420 UV |
| WM06 CADMIUM, TOTAL, BY ICAP | UG/L | 0.592 | UV | 14.0 | V | 0.592 UV |
| WM07 COBALT, TOTAL, BY ICAP | UG/L | 2.47 | UV | 2.67 | UV | 7.98 UV |
| WM08 CHROMIUM, TOTAL, BY ICAP | UG/L | 3.69 | UV | 3.69 | UV | 3.69 UV |
| WM09 COPPER, TOTAL, BY ICAP | UG/L | 1.54 | UV | 2.80 | UV | 1.81 UV |
| WM10 IRON, TOTAL, BY ICAP | UG/L | 29.3 | UV | 33.3 | V | 545 V |
| WM11 MANGANESE, TOTAL, BY ICAP | UG/L | 1.45 | UV | 3.20 | V | 966 V |
| WM12 MOLYBDENUM, TOTAL, BY ICAP | UG/L | 5.89 | V | 23.7 | V | 7.11 V |
| WM13 NICKEL, TOTAL, BY ICAP | UG/L | 6.23 | UV | 9.71 | UV | 6.23 UV |
| WM14 LEAD, TOTAL, BY ICAP | UG/L | 10.4 | UV | 22.9 | V | 41.3 V |
| WM15 ANTIMONY, TOTAL, BY ICAP | UG/L | 17.2 | UV | 17.2 | UV | 17.2 UV |
| WM16 SELENIUM, TOTAL, BY ICAP | UG/L | 40.3 | UV | 40.3 | UV | 40.3 UV |
| WM17 TITANIUM, TOTAL, BY ICAP | UG/L | 4.18 | UV | 4.18 | UV | 4.18 UV |
| WM18 THALLIUM, TOTAL, BY ICAP | UG/L | 36.5 | UV | 1900 | V | 60.3 V |
| WM19 VANADIUM, TOTAL, BY ICAP | UG/L | 2.51 | UV | 2.51 | UV | 2.51 UV |
| WM20 ZINC, TOTAL, BY ICAP | UG/L | 4.07 | UV | 38.6 | UV | 9.68 UV |
| WM21 CALCIUM, TOTAL, BY ICAP | MG/L | 0.530 | UV | 531 | V | 75.4 V |

ANALYSIS REQUEST DETAIL REPORT

ACTIVITY: 9-WLR46

VALIDATED DATA

| COMPOUND | UNITS | 001 | F | 101 | 102 | 301 |
|---|-------|-------|----|-------|----------|-------------|
| WM22 MAGNESIUM, TOTAL, BY ICAP | MG/L | 0.500 | UV | 48.8 | V | 25.3 V |
| WM23 SODIUM, TOTAL, BY ICAP | MG/L | 1.50 | UV | 130 | V | 53.3 V |
| WM24 POTASSIUM, TOTAL, BY ICAP | MG/L | 0.510 | UV | 44.6 | V | 8.19 V |
| WT01 NITROGEN, AMMONIA, TOTAL (NH3+NH4) | MG/L | | | | 0.058 UV | |
| ZZ01 SAMPLE NUMBER | NA | 001 | V | 101 | V | 102 V 301 V |
| ZZ02 ACTIVITY CODE | NA | WLR46 | V | WLR46 | V | WLR46 V |

ACTIVITY WLR46 . DOE RUN-HERCULANEUM SMELTER

THE PROJECT LEADER SHOULD CIRCLE ONE - STORET, AIRS, OR ARCHIVE.

CIRCLE ONE: STORET AIRS ARCHIVE

FINAL DATA REPORT APPROVED BY PROJECT LEADER ON 04/16/99 11:19:40 BY

Bruce Fittell

Facility DOE RUN COMPANY SMELTING DIVISION Facility Ownership THE DOE RUN COMPANY Inspector L. SENA
 Street 881 MAIN ST. Facility Contact JIM LANZAFAME Inspector Phone Ext. 5014
 City HERCUANUM County JEFFERSON Phone (314) 479-5311 SIC code 3339 Primary Media NIPDES
 State MO Zip 63048 Number of Employees 450 Normal Work Hours/Shifts 24 HRS/DAY Date 2/24/91
90 OF WHICH ARE CONTRACTORS 355 DAYS/YR

1. Are there any permits or registrations in the following areas? NPDES (discharge ☒, pretreatment ☐) 404-Wetlands ☐ UIC ☐ UST ☐ PWS ☐ RCRA ☒ TRI ☒ CAA ☒ Other ☐ Describe: HAVE A RUPA GENERATOR # BUT ARE NOT A TSD

2. What does the facility do? PRIMARY LEAD SMELTER & REFINERY

3. What major raw materials are used? LEAD CONCENTRATE (GALENA), COKE, SAND, HEMATITE, (NGA)

COMPRESSED NATURAL GAS

Does facility use more than 200 gallons or 1,500 pounds per month of: Acids ☐ Bases ☒ Ammonia ☐ Chlorine ☐ Chlorinated Solvents ☐

INORGANIC CHEMICALS ☒ Organic Chemicals ☐ Explosives ☐ Fuels ☒ Gases ☒ Solvent-Based Paints ☐ or Solvents ☐ Other ☐

Does facility store on-site more than 100 gallons or 1,000 pounds of: Acids ☒ Bases ☒ Ammonia ☐ Chlorine ☐ Chlorinated Solvents ☐

INORGANIC CHEMICALS ☒ Organic Chemicals ☐ Explosives ☐ Fuels ☒ Gases ☒ Solvent-Based Paints ☐ or Solvents ☐ Other ☐

4. Provide brief process description: PRODUCTION OF PURE LEAD FROM LEAD ORE.

LEAD SULPHATE → LEAD OXIDE

(Check all that apply): Painting/Coating (Water-based ☐, Solvent-based ☐) Printing ☐ Reacting ☒ Formulating ☒ Distilling ☐ Parts Washers/Degreasing (Water-based ☐, Halogenated-based ☐, Non-halogenated-based ☒) Combustion (boiler, furnaces, oxidizers) ☒

Electroplating (Chrome ☐, Other ☐) Electroless plating (Type ☐)

5. Describe each waste generated by the facility:

Is the waste hazardous?

| Waste Name | Generation Process | Quantity/Month | Final Disposition of Waste | How Long Stored | No | Yes | Don't know |
|--|----------------------|-----------------------|----------------------------|-----------------|--------------------------|-------------------------------------|--------------------------|
| <u>SAFETY KLEEN SOLVENT PARTS WASHER</u> | <u>CALL</u> | <u>99 LBS/MO</u> | <u>RECYCLED</u> | <u>1 MONTH</u> | <input type="checkbox"/> | <input checked="" type="checkbox"/> | <input type="checkbox"/> |
| <u>REFRACTORY BRICK</u> | <u>EQUIP. REPAIR</u> | <u>69.325 TONS/MO</u> | <u>TSD LANDFILL</u> | <u>90 DAYS</u> | <input type="checkbox"/> | <input checked="" type="checkbox"/> | <input type="checkbox"/> |
| <u>MOST OF THIS LOSS IN 2 MONTHS. FAILURE WAS 1988 AVERAGE</u> | | | | | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| <u>THIS AVERAGE WAS UNUSUALLY HIGH BECAUSE THEY REPAIRED</u> | | | | | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| <u>THE BRICK IN 1 TONER DURING A REPAIR</u> | | | | | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |
| | | | | | <input type="checkbox"/> | <input type="checkbox"/> | <input type="checkbox"/> |

ENVIRONMENTAL JUSTICE (EJ)

1. What type of area is the facility located in? Industrial ☐ Business ☐ Residential ☒ Rural ☐

Does the area appear to be run down, poorly maintained, or have many abandoned and dilapidated properties? No ☒ Yes ☐

2. What is the estimated income level of the residents in the area that may be impacted by the facility? Low ☒ Moderate ☒ High ☐

3. How close are the nearest normally occupied properties (houses, apartments, schools): <100' ☐ 100-1000' ☒ 1000'-1 mile ☐ >1 mile ☐

NATIONAL POLLUTION DISCHARGE ELIMINATION SYSTEM (NPDES), UNDERGROUND INJECTION CONTROL (UIC), PUBLIC WATER SUPPLY (PWS)

1. How are wastewaters handled? None ☐ On-site Treatment ☒ Municipal Sewer ☐ Storm Sewer ☐ Surface Water ☒ Septic ☐ Disposal Well ☐ Land ☐

Process wastewater → ☐ ☒ ☐ ☐ ☒ (TREATED) ☐ ☐ ☐

Non-contact wastewater → ☒ ☐ ☐ ☐ ☒ ☐ ☐ ☐

Sanitary wastewater → ☐ ☐ ☒ ☐ ☐ ☐ ☐ ☐

Comments: SURFACE WATER IS TREATED ON SITE NON-CONTACT BEARING COOLING WATER IS OUTFALL 003

2. Did you see any wastewater discharges not identified by the facility? No ☒ Yes ☐ Location of discharge: _____

Appearance of discharge: _____ (PHOTO ☐)

3. What is the source of the facility's drinking water? Rural/Municipal ☒ Private well ☐ River ☐ Other ☐

4. Is the facility's water source protected with a backflow prevention device? No ☐ Yes ☒ Don't know ☐

WETLANDS (CWA - Section 404)

1. Did you see any streams, rivers, ponds, lakes, or temporarily wet areas being (or have been) disturbed by filling, dredging, channelizing, damming, excavating, gravel removal, etc.? No ☒ Yes ☐ Don't know ☐ Describe/locate: _____ (PHOTO ☐)

CLEAN AIR ACT (CAA)

1. Did you see any visible smoke or dust emissions? (non-steam) No ☒ Yes ☐ Source: _____ Time: _____ (PHOTO ☐)
2. Did you see any dust leaving the property? No ☒ Yes ☐ Source: _____ Time: _____ (PHOTO ☐)
3. In the past 2-3 years, has the facility modified or installed any new air emission points? No ☐ Yes ☒ Describe: INSTALLED A NEW ARE-
EATER FOR ACID PLANT, SMALL DIRECT SMELTING FURNACE Was a permit obtained? No ☐ Yes ☒ Permit No: 1998-D-7-005 + 1998-D-8-037

4. Are there stationary air conditioning or refrigeration units that contain? < 50 lbs refrigerant/unit ☐ > 50 lbs refrigerant/unit ☐ Both ☒ None ☐
Are these units: Self-serviced? ☐ Contract Serviced? ☒ → Service Company: SHEET METAL CONTRACTORS

5. Are motor vehicle air conditioning systems: Self-serviced? ☐ Contract Serviced? ☒ → Service Company: LOCAL AUTO SHOP None ☐
3 COMPANY VANS

RESOURCE CONSERVATION AND RECOVERY ACT (RCRA) AND UNDERGROUND STORAGE TANKS (UST)

1. EPA Hazardous Waste Identification Number? No ☐ Yes ☒ → # MDMD6266373 : Generator Size LARGE QUANTITY
2. Does facility: Treat ☐ Burn ☐ Landfill ☐ or use Surface Impoundments ☐ for on-site hazardous waste management? No ☒
3. Did you see large numbers of drums (>15) of unknown materials stored in an "abandon-like" manner? No ☒ Yes ☐
Describe: _____ (PHOTO ☐)
4. Did you see any hazardous waste containers, drums, or tanks leaking? No ☒ Yes ☐
Describe: _____ (PHOTO ☐)
5. Did you see any signs of spills or releases (e.g., dead or stressed vegetation, stains, discoloration)? No ☒ Yes ☐
Describe: _____ (PHOTO ☐)
6. Did you see any chemical, industrial, or waste handling practices that concerned you (consider access to children and public)? No ☒ Yes ☐
Describe: _____ (PHOTO ☐)
7. Does facility have any past or present underground storage tanks that contain petroleum, used oil, or hazardous substances? No ☒ Yes ☐
8. Does facility have any underground fuel storage tanks for emergency generators? No ☒ Yes ☐

EMER. PLANNING & COMMUNITY RIGHT TO KNOW ACT (EPCRA), TOXIC SUBSTANCES CONTROL ACT (TSCA) & PCB's (Polychlorinated Biphenyls)

1. Have Toxic Chemical Release Forms (Form R) been submitted under Section 313 of EPCRA? No ☐ Yes ☒
2. Have Hazardous Chemical Inventory Forms (Tier II) been submitted to local Emergency Planning Committees or fire departments? No ☐ Yes ☒
3. Does facility import or manufacture a chemical substance? No ☐ Yes ☒ Describe type and intended use: MANUFACTURE H₂SO₄
4. Does facility have equipment containing PCB's > 500 ppm in storage or service - that is leaking ☐ not labeled ☐ or not registered ☐? No ☒

SPILL PREVENTION CONTROL AND COUNTERMEASURE PLAN (SPCC)

1. Does facility have above ground oil (petroleum, synthetic, animal, fish, vegetable) tanks, with an aggregate volume > 1320 gallons? No ☐ Yes ☒
Is there an SPCC Plan? No ☐ Yes ☒ Is there secondary containment? No ☐ Yes ☒
Is oil leaking and threatening to reach waters of the State or U.S.? No ☒ Yes ☐ Describe: _____ (PHOTO ☐)

FEDERAL INSECTICIDE, FUNGICIDE, AND RODENTICIDE ACT (FIFRA)

1. Does the facility manufacture, repackage, or apply pesticides? No ☒ Yes ☐
Are rinsates handled in an environmentally sound manner? Yes ☐ No ☒ → Describe: _____ (PHOTO ☐)
2. Do workers use personal protective equipment (gloves, long sleeve shirts, coveralls) when mixing, loading, or applying? No ☐ Yes ☒

* PLEASE TAKE PHOTOS TO DOCUMENT POTENTIAL PROBLEMS

APR 23 1999

MEMORANDUM

SUBJECT: Transmittal of Inspection Report - Water

FROM: Lorenzo Sena, Environmental Protection Specialist
EMWC/ENSV

TO: Cynthia Hutchison, Compliance Officer
NFMB/WWPD

This memorandum transmits the following inspection report conducted by the Environmental Services Division:

| | |
|--|--|
| Type: Compliance Biomonitoring Inspection - Acute | Inspection Date: 02/22/99 |
| Facility Name: Doe Run Herculeaneum Smelter | Facility I.D. Number: NPDES-MO-0000281 |
| Address: 881 Main Street Herculeaneum, MO 63048 | Activity Number: WLR46 |
| Facility Activity: Lead ore converted to lead metal | SIC Code: 3339 |
| Environmental Justice: Was the inspection conducted in a potential EJ area? No | |
| Multimedia: Screening checklist completed? Yes (Or: Level C multimedia inspection completed involving Air & RCRA.) No | |
| Preliminary Findings: 1. Holding time for pH analysis is greater than 15 minutes. 2. SIC code for facility is 3339, permit is incorrect, SIC code on permit is 3332. | |
| Comments: | |

Also, we request that you complete and return the attached Satisfaction Survey.

Attachments

cc: Mary Tietjen-Mindrup, NFMB/WWPD, w/o attachments

L.Sena:vs:04/20/99

EMWC

Lizell
4/20/99

EMWC

4/20/99